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SUNNYVALE, CALIFORNIA

FINAL REPORT

BINDER-FILLER

INTERACTION IN

ENERGETIC POLYMERS

ONR CONTRACT NOO014-83-C-0271

MARCH 31. 1985

R. H. MARTINSON



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ACKNOWLEDGEMENT

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The materials tested were all prepared at the Naval Weapons Laboratory, China Lake under the direction of Dr. Rena Yee and Dr. Eugene Martin who also provided advance copies of their own data. Dr. R. S. Miller of the Office of Naval Research served as contract monitor and provided several helpful suggestions.

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INTRODUCTION

/The objective of this study was to measure the kinetics of the dewetting process in a variety of filled energetic polymers being studied as candidates for improved solid rocket and gun propellants in the Office of Naval Research ONR Power Program under the direction of Di. R. S. Miller (Code 432). The experimental method used was ultrasonic and electromagnetic wave scattering (SAXS) which had been previously developed at the Lockheed Palo Alto Research Laboratory as valuable tools for characterizing strain-induced internal damage in tactical solid propellants.

As many experimental compositions as were available in the requisite amounts from the Naval Weapons Center, China Lake, were to be investigated, with 4 being the agreed upon minimum number. Actually, experiments were performed on a total of 9 different model compositions, although both ultrasonic and SAXS experiments were not carried out on every composition due to the lack of suitable specimens.

The motivation for this investigation was the "hot-spot" theory of initiation. It has been known for some time that porous explosives are much more sensitive to initiation by impact than are fully dense cast explosives. Empirical correlations between impact sensitivity and mechanical properties have also been developed. In the propulsion industry, it is widely believed that the devetting process is central to the whole question of what determines the mechanical properties of heterogeneous propellants.

The energetics of wetting of single crystal RDX and HMX by energetic binders had been studied at NWC by Dr. Rena Yee prior to the commencement of this investigation [1]. It was hoped that

by studying the dewetting process in detail. correlations between surface energetics and dewetting behavior would be obtained.

Specific questions addressed in the present work are:

- What is the relation, if any, between the particle size of RDX and the voids created by dewetting?
- 2) Does the number density of voids caused by strain vary with solids loading in the same binder?
- What is the influence of plasticizers on the size and number density of voids?
- What is the relative number density of voids in compositions that fracture at different strains? The size?
- 5. What do the growth curves of voids look like as functions of strain?

By combining SAXS and ultrasonic wave scattering (UWS) data with existing theories of scattering, partial answers to these questions can be derived. It is for the lack of adequate scattering theory rather than for the lack of experimental precision that complete and definitive answers cannot always be given.

MATERIALS

The materials and test specimens for this experimental investigation were prepared at the Naval Weapons Center, China Lake, under the direction of Dr. E. C. Martin. The model materials are described in Ref. [2] and listed in Table 1.

<u>,</u>	COMMENTS		Samples highly absorbing - too thick. Tensile &	relaxation runs not successful.						Tensile runs	Tensile runs	LMSC-FO	35789
	DATES	12/02/83	12/02/83, 12/04	12/03/83, 12/04	03/25/85, 03/26 11/02/84	03/25/84			11/02/84	10/19/84 01/17/85	11/02/84 01/17/85	11/05/84	11/05/84
<u>.</u>	EXPERIMENTS RUN	SAX	SAX	SAX	SAX UMX	SAX	Chemical Analysis	Chemical Analysis	UMS	UMS	UMS SAX	CMS	CMS ,
	COMPOSITION	R45M-1PD1 75% RDX (E & C)	R45M-IPDI 80% RDX (E & Screened A)	R45M-IPDI 75% RDX (E & Screened A?)	18.75% TMETN + 6.25% GAP + 75% RDX	36% R43M-IPDI 64% RDX (Class E) cast	(screened)		4.95% GAP + TMETN 45% Class E RDX; 30% Class A RDX	26.72% GAP + 4.68% N-100 41.13% Class E RDX 27.42% Class A RDX	9.18% GAP + 2.09% N-100 +22.63% BTTN 39.54% Class E RDX 26.36% Class A RDX	PBX 29.01% Class A RDX 43.51% Class E RDX	23.2% R45M-IPDI 45% Class E RDX cast 30% Class A RDX
	UEST GNATION	BLX-1	BLX-2	BLX-3	BLX-4-6	BLS-1	Class A ROX	Class E RDX	BLX-4	BLX-8	BLX-9	BLx-6	BLX-11
DATE SHIPPED	TRUM IN	11/17/83	11/17/83	11/17/83	03/29/84	03/29/84	03/29/84	03/29/84	07/11/84	07/17/84	07/17/84	07/17/84	07/17/84

TABLE 1. Summary of Materials, Compositions, and Experiments Performed.

Because of the physical nature of some of the materials such as brittleness or stickyness, NWC was not always able to prepare test specimens in the geometries specified by LMSC. For instance, the cutting of SAXS specimens into thin uniform slices whose major faces were accurately parallel did not prove possible. The limited amount of material available meant that UWS specimens usually turned out to be smaller than is desirable for such experiments. It was decided jointly by NWC and LMSC to accept the existing limitations on purity, shape, and numbers of test specimens in order to avoid delaying the experimental program. There is no doubt, however, that more consistent experimental data could have been obtained with better test specimens.

CHEMICAL ANALYSES

During early SAXS experiments it was noticed that the x-ray absorption of samples received from NWC appeared to be higher than calculated from the nominal elemental composition. It was therefore decided to analyze some of the specimens chemically.

Results of chemical analyses performed on BLX-1 and neat RDX are shown in Tables 2 and 3. The analyses were done by igniting the samples in air and weighing the residues which were then subjected to semiquantitative emission spectrographic analyses. It was believed that major heavy elemental impurities with the exception of Bismuth would be found in RDX rather than in the binder. Therefore, samples of both Type A and E RDX were obtained from NWC and analyzed chemically. The results are shown in Table 3.

It was, indeed, found that most of the impurities are contained in the RDX. Bismuth-containing chemicals are

Weight-percent residue after ignition - 0.011%.

Semiquantitative emission analysis of ash as oxides (in percent):

Mg		.0022
Si	•	.0020
Bi		.0018
Fe	. "	.0013
Na		.0009
Sn	4	.0005
Cr	-	.0007
Ca		0004
В		.0004
Pb		.0002
Cu		.0002
Ag		.0062
Ti		.0001
Mn	•	.0001
Al	¥	.0001
		.011

TABLE 2. Results of Chemical Analysis of BLX-1 Material.

Two samples of RDX received from NWC were air oxidized by thermal decomposition in platinum crucibles. The weight of each sample was nearly 4 grams. The ash was subjected to emission spectrographic analysis.

Sample		<u> </u>	<u>E</u>
Percent residue	1	·	
after ignition	•	$0.0074 \pm .0005$.0072 ± .0005
Spectrographic	_		
analysis (as oxides)	Mg	17 ppm	18 ppm
-	Si	16	17
•	Fe	13	13
	Ca	7	. 7
	Cr	4	4
	Ni	4 .	4
	Cu	4	4
	Pb	2	0
	Na	. 1	1
	Ti	2	2
	Zn	. 1	(1
	Al	2	2
	Sn	1	1.
	Ag	1	(1)
	В	1	1

TABLE 3. Results of Chemical Analyses of Class A and E RDX Received from NWC.

deliberately added to the mixes. Even though the total amount of metals in BLX-1 (and probably in the other mixes) is only about 100 ppm, the x-ray absorption is affected significantly by these impurities.

PARTICLE SIZE DISTRIBUTIONS

NWC provided the following information regarding the particle size distributions of RDX used in the mixes:

Class E RDX -	Average diameter:	4 microns
Class C RDX -	80% > 150μ 20% < 150μ	

Screered Class A RDX

Microns		Weight %
- 20		9.5
20 - 40	4	8.0
40 - 60	,	11.8
60 - 80		17.7
80 - 100	·	23.0
100 - 120		18.2
120 - 150		11.8

TABLE 4. Particle Sizes of RDX Used in the Materials.

ULTRASONIC WAVE SCATTERING (UWS) EXPERIMENTS

The basic methodology of ultrasonic testing developed at LMSC for measuring internal mechanical damage in propellants and other filled polymers is described in References E3J and E4J.

Briefly, pulsed ultrasonic wave transmission measurements are made as the test specimen is deformed mechanically. From measurements of wave speed changes, the volume dilatation is calculated according to existing propagation theory for porous materials. The average scatterer size is then calculated from the measured attenuation and dilatation by invoking the theory of Rayleigh scattering.

Over a period of several years of experimentation at LMSC it has been found that the correlation between ultrasonically measured dilatation and dilatation measured directly is usually very good for a variety of filled polymers and propellants. However, it has also been found that Rayleigh scattering theory yields a mean scatterer size that is substantially larger than is reasonable, and that the number density of scatterers is smaller than seems reasonable. Indeed, in those few cases in which the frequency dependence of ultrasonic attenuation has been measured in strained propellants, it has been found that the attenuation varies approximately as the square of the frequency, rather than as the fourth power as required by Rayleigh's law. This observation shows that pure Rayleigh scattering probably is not the only source of incremental attenuation in strained solid propellants. Accordingly, the vacuoule sizes derived from applying Rayleigh scattering theory to ultrasonic attenuation experiments cannot be regarded as quantitative, particularly in the usual case when a distribution of scatterers contributes to the total attenuation. Nevertheless, for lack of other useful scattering theory, we have in this report included calculations

of the mean size and number density of vacuoules according to Rayleigh theory.

The basic measurements taken during the ultrasonic experiments are:

- 1. Uniaxial extension of the specimen.
- 2. Transverse contraction of the specimen.
- 3. Change in transit time of the transmitted ultrasonic waves.
- 4. Change in attenuation of the transmitted ultrasonic waves.
- 5. Ratio of the speeds of shear waves and longitudinal waves in undeformed specimens.

A block diagram of the ultrasonic measurement apparatus is shown in Figure 1 and a photograph of the transducer holder mounted on a propellant specimen is shown in Figure 2.

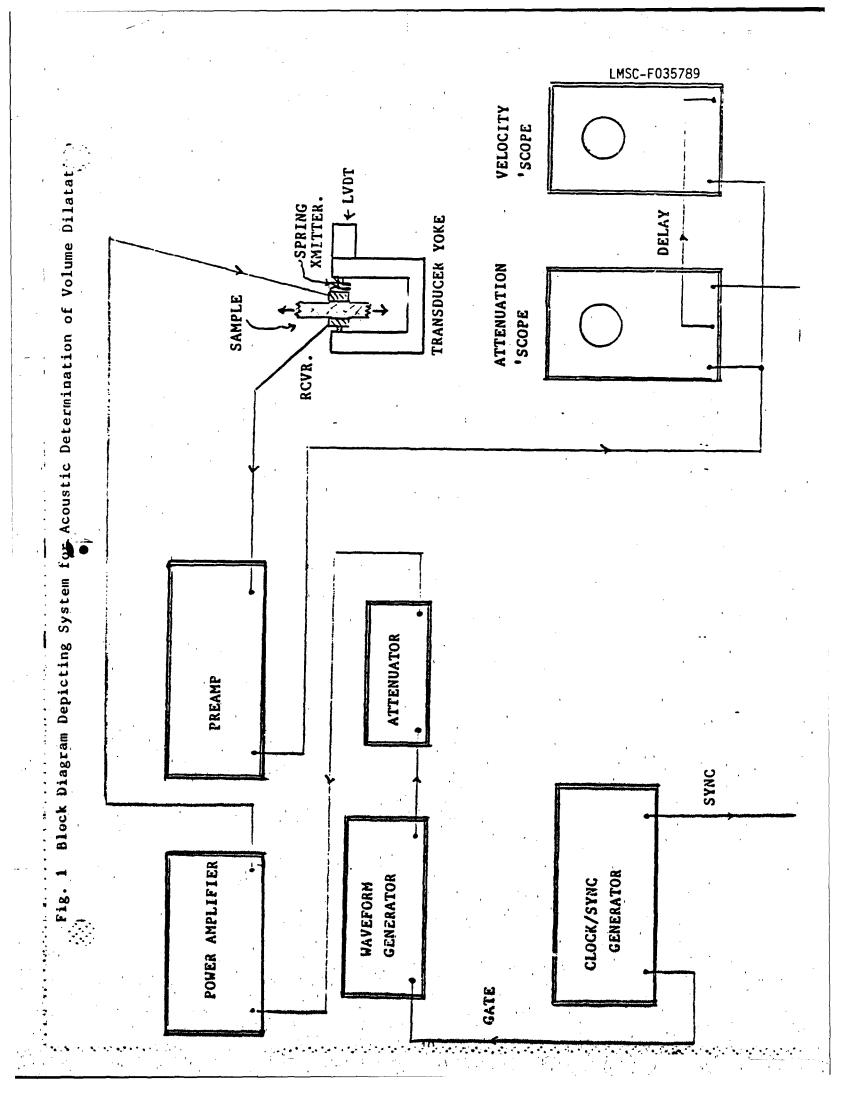
Two basic equations derived in Reference [3] have been used to reduce the ultrasonic data. These equations are:

$$\frac{\Delta \underline{V}}{\overline{V}_{o}} = \frac{1}{b} \begin{bmatrix} \frac{\Delta \underline{C}}{\overline{C}_{o}} \\ \frac{1}{1} - \frac{\overline{\Delta}\underline{C}}{\overline{C}_{o}} \end{bmatrix}$$
(1)

$$\Delta \alpha = \frac{1}{6} \varsigma k^4 a^3 \frac{\Delta V}{V_0} \tag{2}$$

where
$$b = 8(1 - 32v_0)$$

$$k = \frac{2\pi}{3}$$



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Fig. 2 Ultrasonic Transducers Mounted on Propellant Specimen in Tensile Test Machine

and the function g is a known function of Poisson's ratio υ_{o} while $\frac{\Delta C}{C}$ is the fractional change in longitudinal wave speed and $\frac{\Delta V}{V_{o}}$ is the fractional dilatation and λ is the ultrasonic wave length.

Plots of the ultrasonic data for BLX-4, BLX-6, BLX-8, BLX-9, and BLX-11 are included in Appendix 1. Each data set consists of 6 plots which show:

- 1. The Joad-strain curve.
- 2. Relative change in sound speed.
- 3. Relative change in attenuation.
- 4. Calculated volume dilatation.
- 5. Calculated void radius.
- 6. Calculated void number density.

Included also in Appendix 1 are plots of volume dilatation data obtained from NWC and a table of longitudinal and transverse wave speeds for 5 undeformed specimen compositions.

The ultrasonic data in Appendix 1 appear remarkably similar for all the materials tested. Since the RDX particle distribution and solids loading are essentially the same for BLX-4, 6, 8 and 11, this result is to be expected. Apparently the binder affects the sound speed variation and the incremental attenuation only slightly. Of course, the binder composition affects the stress-strain curve, particularly the yield point and the strain to break which increases with plasticizer content.

It is troubling that there is not better quantitative agreement between the dilatation measured at NWC and the dilatation derived from ultrasonic measurements. Part of the reason for this is probably the "squat" specimens that were used for the ultrasonic measurements - the strain distribution in

these specimens was probably not one of uniaxial tension over the whole length of the active area of transducer.

Within the limits of Rayleigh scattering theory, the voids appear to grow rapidly to a mean radius of 0.03 cm and remain essentially of constant size thereafter as the specimen extends. The number density of voids increases with strain as is shown on the plots.

The quantitative values of void radius and number density are likely to be in error in these plots, but the qualitative nature of the variation with strain may be correct. It is, in any case, similar to what we have observed in tactical propellants. It would take a major theoretical effort to improve the scattering theory in order to derive quantitative void distribution data from ultrasonic measurements on these materials.

SMALL ANGLE X-RAY SCATTERING (SAXS) EXPERIMENTS

The objective of the SAXS investigation was to study the kinematics of void nucleation in the model materials when these were mechanically deformed. Since it was expected that rapid events (seconds) would be encountered, it was decided to use the Stanford Synchrotron Radiation Laboratory's SAXS facilities which generate x-ray fluxes 10² to 10³ times more intense than conventional x-ray tubes. It was hoped that the scattering data describing void nucleation processes could be acquired fast enough on this high flux facility to yield acceptable signal to noise ratios with an integration time of 0.1 seconds. Prior experiments at SSRL had demonstrated that this could be done for strong scatters [5].

The basic SAXS experiment consists of deforming the sample in a monochromatic x-ray beam and registering the angular dependence of the scattered intensity with an x-ray detector. It is necessary that the detector be sensitive enough and have a fast enough response time to register time dependent events on the time scale of interest.

A system that appeared to meet these requirements had been designed and built at SSRL on Beam Line I by Dr. G. B. Stephenson. This system, which is described in detail in Dr. Stephenson's dissertation [6], was modified by the addition of a specimen deformation device to yield a complete apparatus for the SAXS study of deformed polymers. The modified system is described in Reference [7].

Initial SAXS experiments on BLX-1, BLX-2 and BLX-3 were run at SSRL during the time period 12/2 to 12/4, 1983. On examining the data it was found that all these specimens absorbed x-rays to a much greater extent than had been calculated, resulting in poor signal to noise ratios. This observation prompted the chemical analyses the results of which have been reported above.

On the basis of these results, new specimens were designed which were about 30% thinner. Acceptable x-ray transmission was observed for the specimens BLX-4-6 and BLS-1 during the run period 3/25 to 3/26, 1984. However, excessive detector noise, possibly caused by radiation damage to the detector appeared in these data. The data for BLX-4-6 are shown in Appendix II.

The data in Appendix II represent differences between the scattering from the undeformed sample and the deformed sample corrected for changes in x-ray absorption. The relative intensity is plotted on logarithmic scale vs. the length of the

scattering vector $\frac{4\pi}{\lambda}\sin\frac{\Theta}{2}$, where Θ is the scattering angle and λ is the x-ray wavelength.

We have interpreted the SAXS data according to the theory of Guinier, according to which

$$I(h) = C V_a V_p (\Delta p)^2 e^{-\frac{R_q^2 h^2}{3}}$$

In this equation

I(h) = x-ray intensity distribution

V_ = mean atomic volume

C = concentration of voids

V_p = volume of particles (or voids)

Δp = the difference in local average electron distribution inside and outside the particle

(or void)

 R_{q} = radius of gyration

 $h = \frac{4\pi}{\lambda} \sin \frac{\Theta}{2}$

 $\lambda = x$ -ray wavelength

Θ = scattering angle.

For spherical particles with radius Ra,

$$R_{\alpha}^{2} = \frac{3}{5} R_{s}^{2}$$

The theory of Guinier applies strictly only to dilute concentrations of monodisperse scatterers. However, it is widely used as an approximation to interpret experimental data even in the case that distributions of particles are present. In this case it is believed to yielu semi-quantitative results for R_q .

The general results of our SAXS experiments are that a distribution of voids with diameters from 150 to 800A form near the yield points of the model compositions BLX-4, BLX-C, and

BLX-9. There is an apparent fluctuation of the concentration as the specimen is extended further, which we interpret as evidence that the concentration of voids is heterogeneous. The reason for this is that as the specimen is extended in the x-ray beam, different volume elements translate into and out of the beam. After rupture, the scattered intensity decays with time.

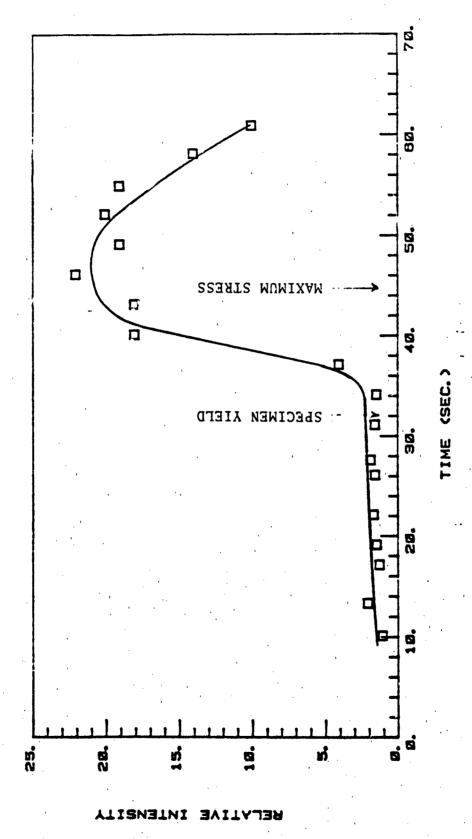
A typical plot of the variation of scattered intensity for a tensile test of BLX-4 is shown in Figure 3. Because the distribution function of the voids cannot be measured by SAXS, it is not possible to quantify the void concentration from data such as that presented in Figure 3.

SUMMARY AND CONCLUSIONS

A combined ultrasonic wave scattering and small angle x-ray scattering study of several model compositions containing RDX has been carried out. The dewetting behavior of these materials has been measured as a function of time and strain by subtracting the initial scattering from the scattering of deformed samples. The residual scattering, both of ultrasonic waves and x-rays, has been attributed to voids.

By combining the results of SAXS and UWS, it is concluded that a wide distribution of void sizes (150A to 0.03 cm) are created by mechanical strain in these materials. The concentration of voids increases with strain from the yield point to rupture, while the sizes appear to remain almost constant.

The details of the void distribution and size are not affected by binder composition and plasticizer content.



Variation of Scattered X-ray Intensity With Time During Tensile Test of BLX-4

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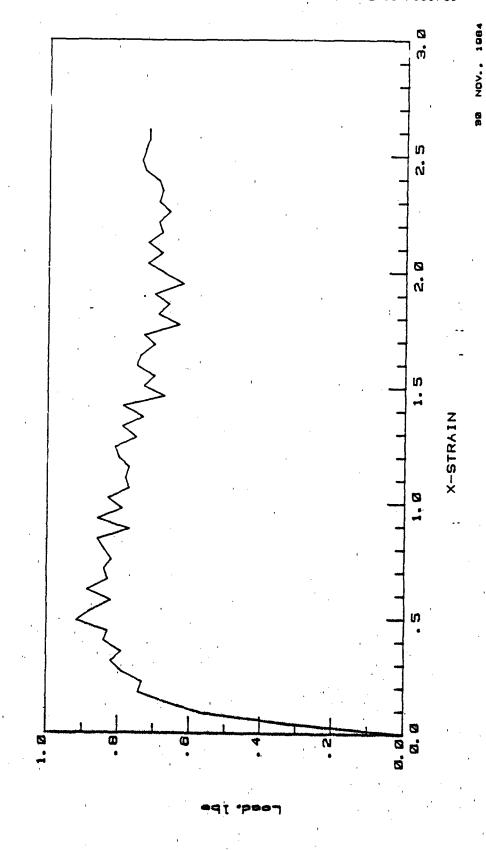
APPENDIX 1

ULTRASONIC DATA

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ULTRASONIC DATA FOR BLX-6

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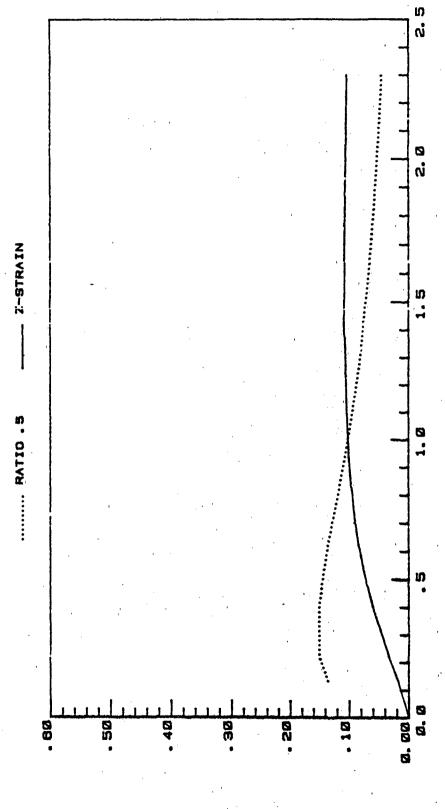


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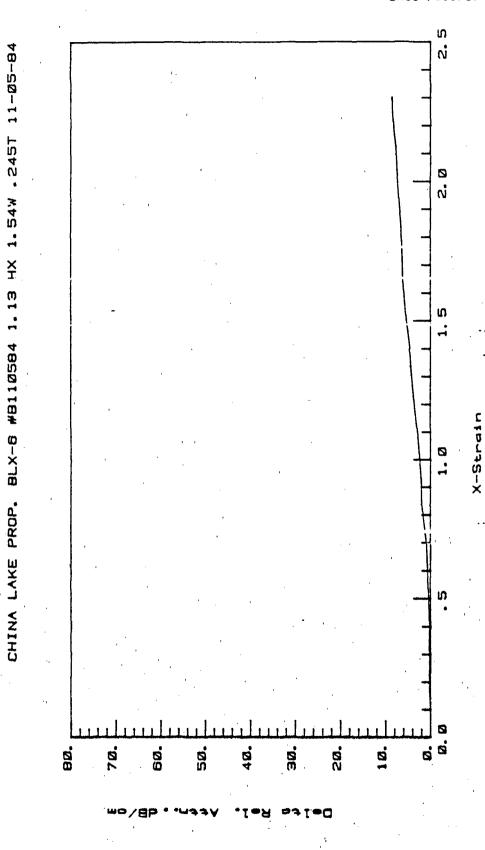
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X-STRAIN

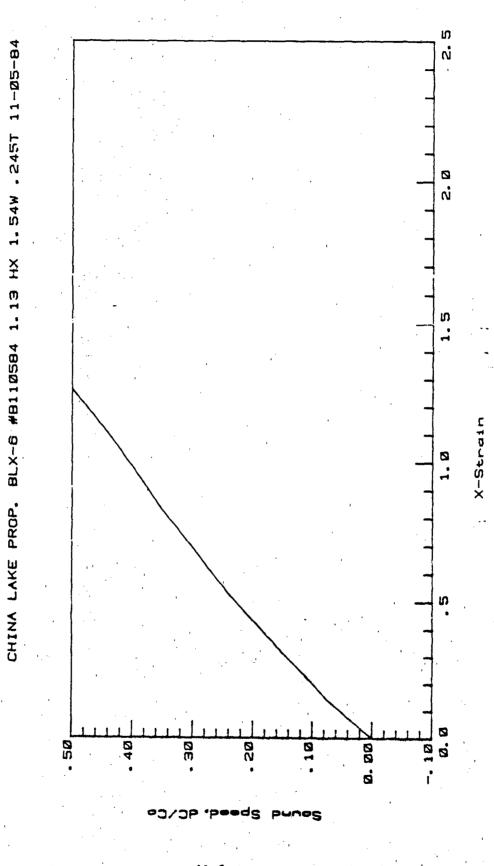
LABORA TORY CHINA LAKE PROP. BLX-8 #B110584 1.13 HX 1.54W . 245T 11-05-84 CKHEED MATERIALS ANALYSIS

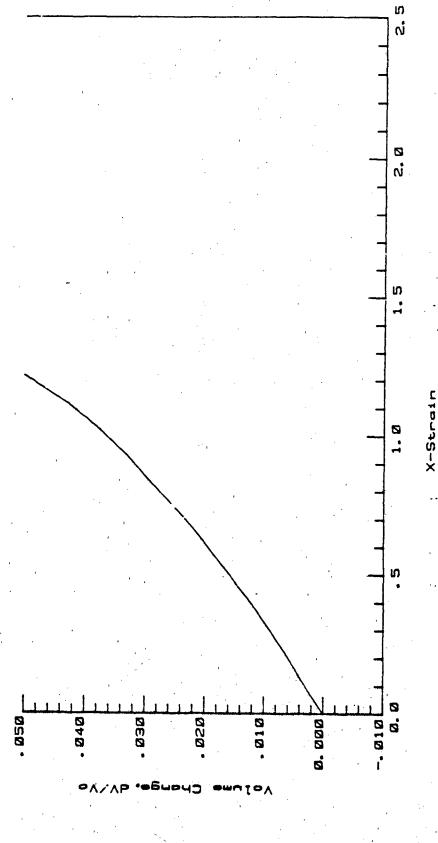


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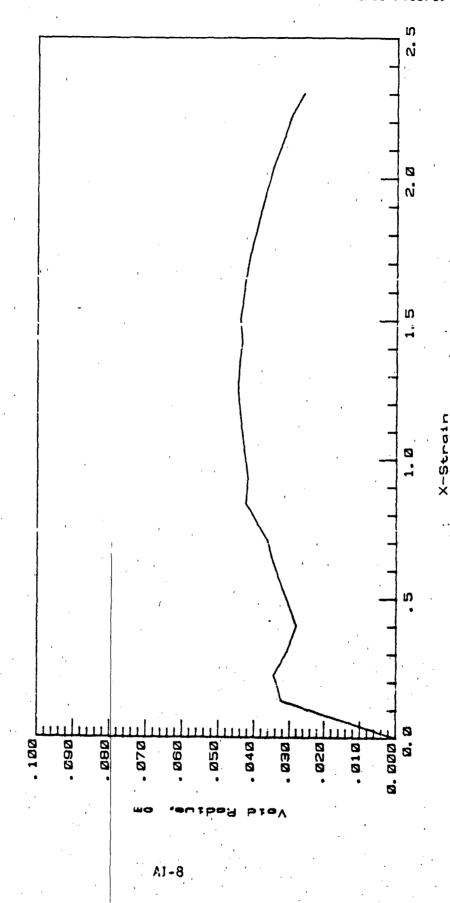


LABORA TORY LOCKHEED MATERIALS ANALYSIS

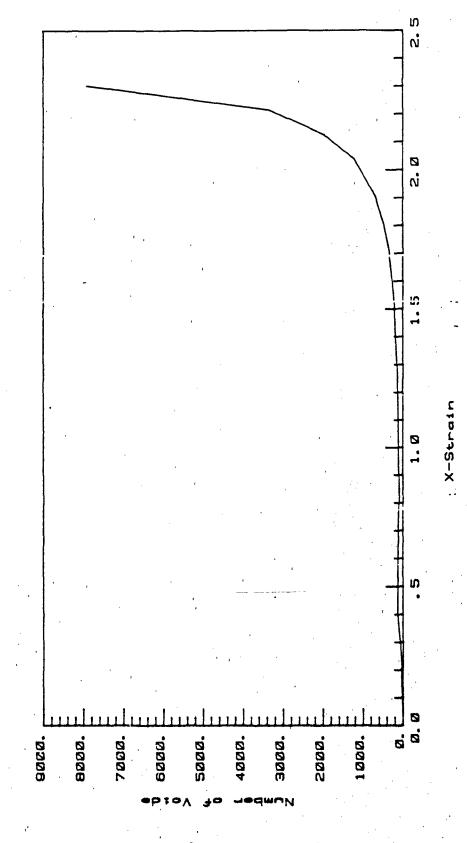




CHINA LAKE PROP. BLX-6 #B110584 1.13 HX 1.54W .245T 11-05-84 -OCKHEED MATERIALS ANALYSIS LABORATORY



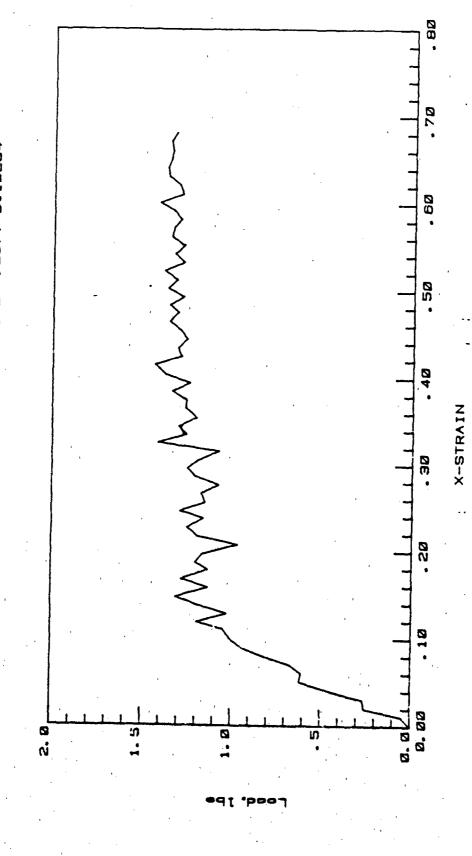
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A1-9

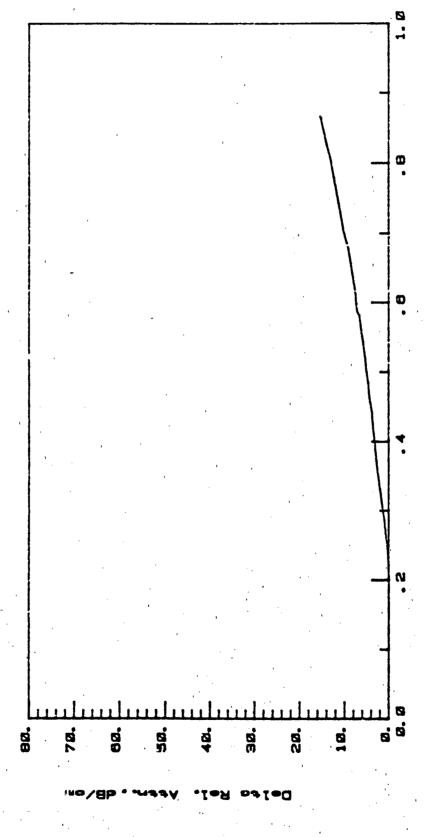
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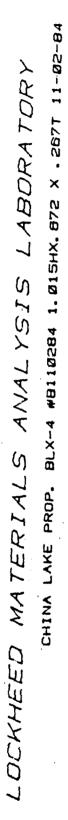
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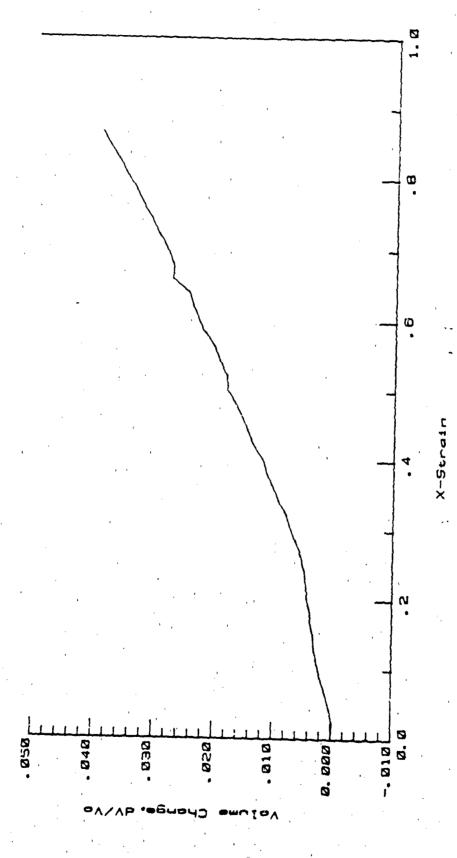


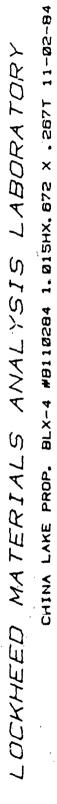
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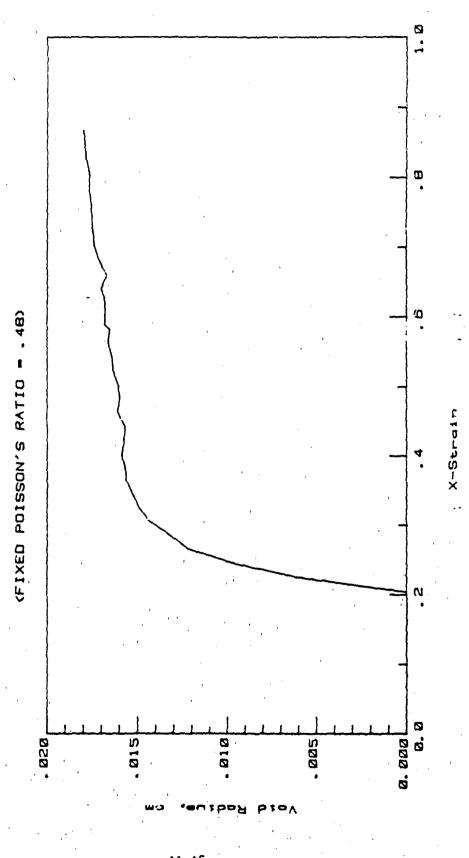
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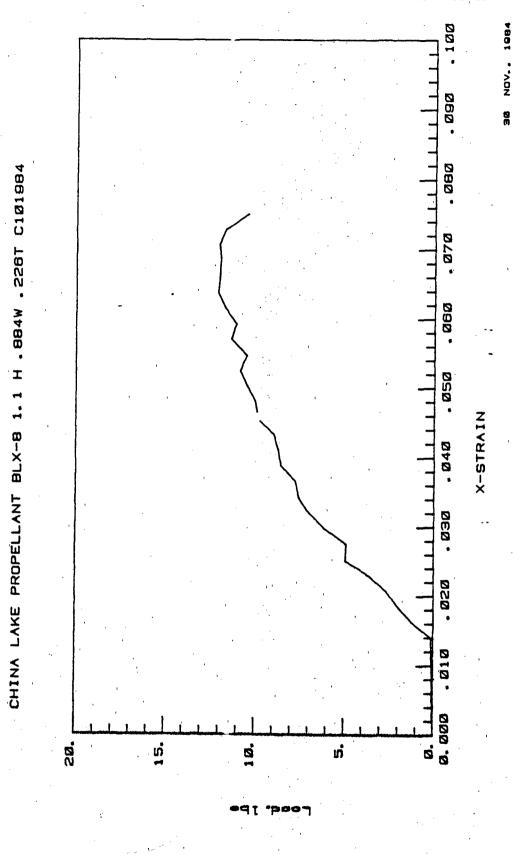




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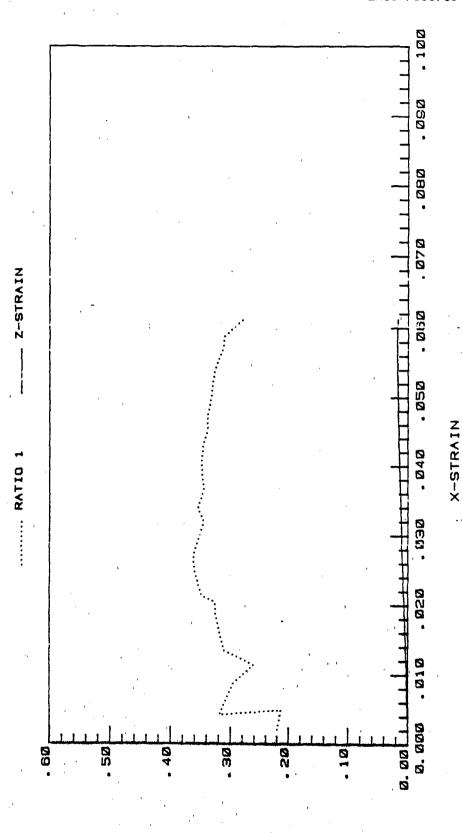
ULTRASONIC DATA FOR BLX-8

LOCKHEED MATERIALS ANALYSIS LABORATORY



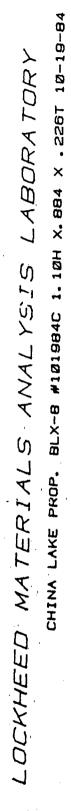
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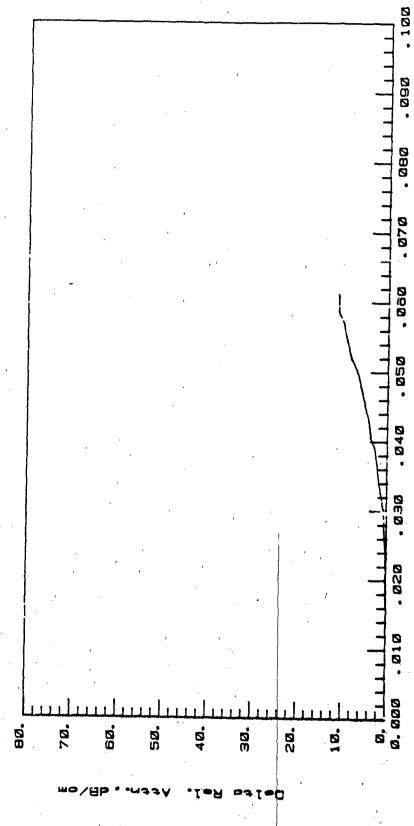
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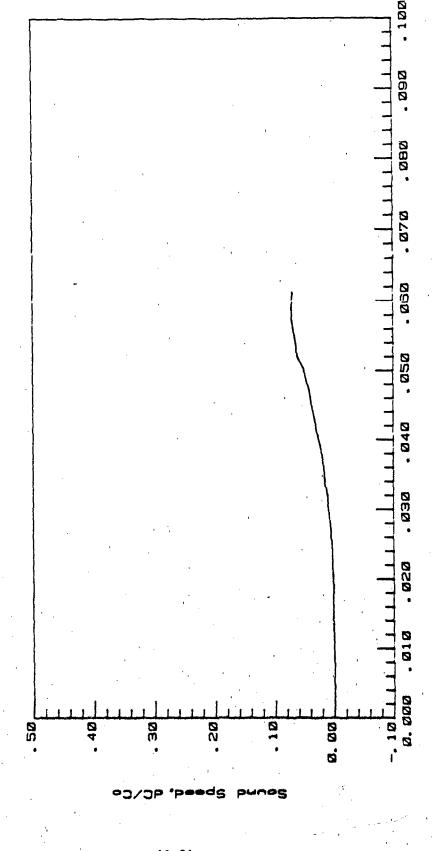
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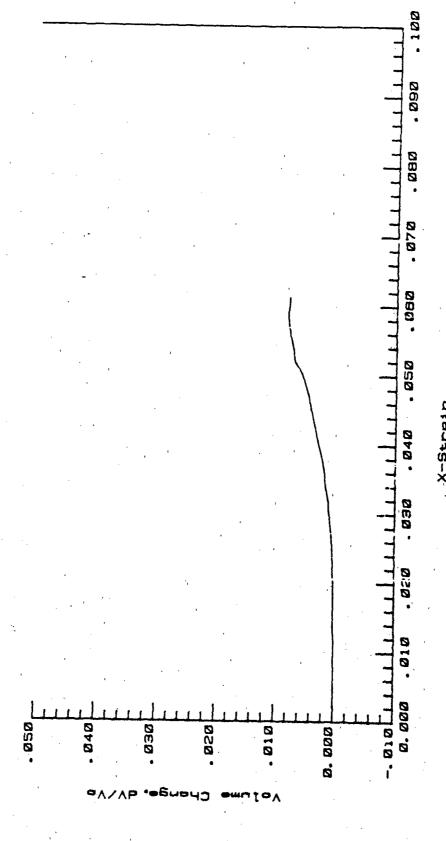
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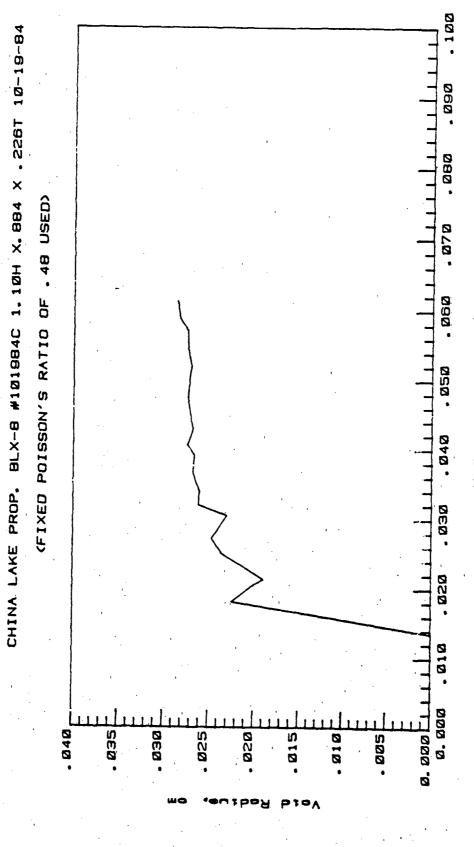
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LOCKHEED MATERIALS ANALYSIS LABORATORY

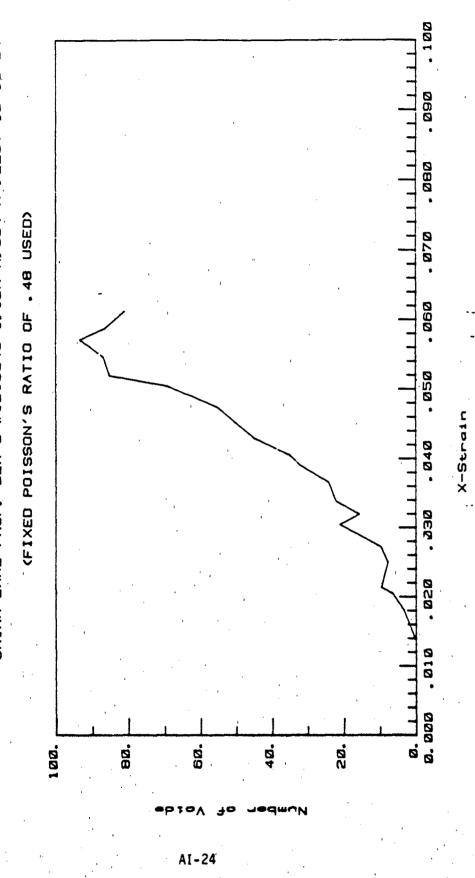




LOCKHEED MATERIALS ANALYSIS LABORATORY

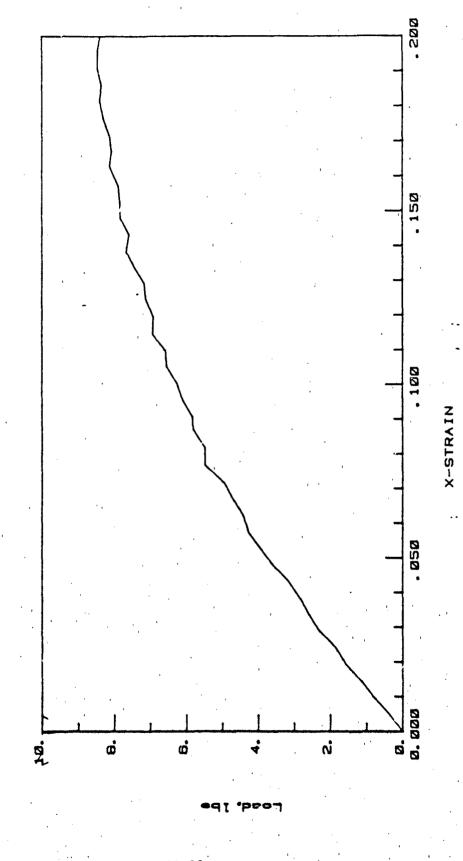


CHINA LAKE PROP. BLX-8 #101884C 1.10H X.884 X . 226T 10-19-84 LOCKHEED MATERIALS ANALYSIS LABORATORY



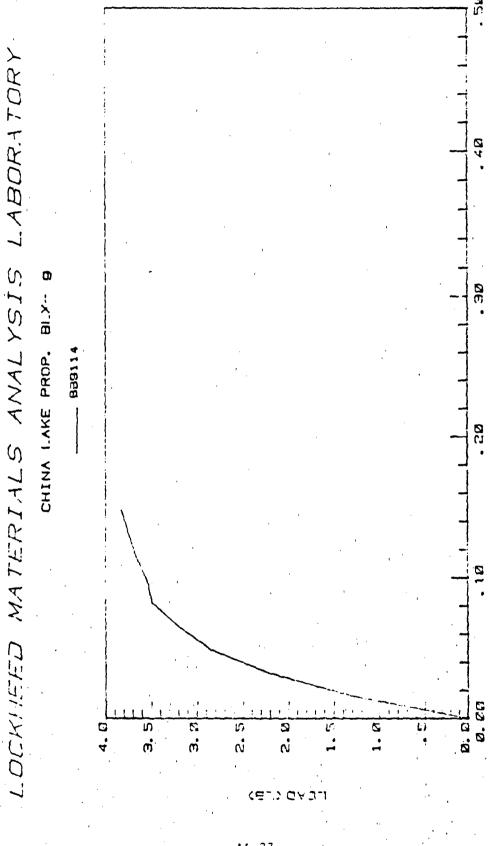
ULTRASONIC DATA FOR BLX-9

LOCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE PROPELLANT BLX-9 1.05H .75W .277T A110284



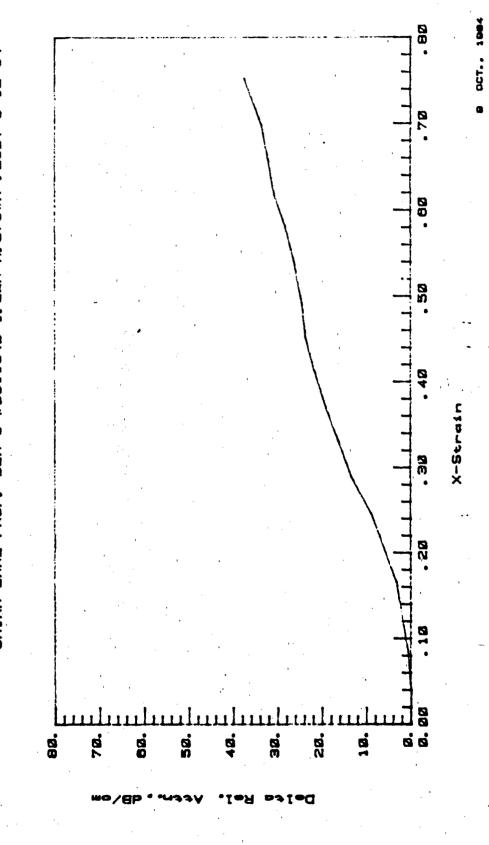
AI-26

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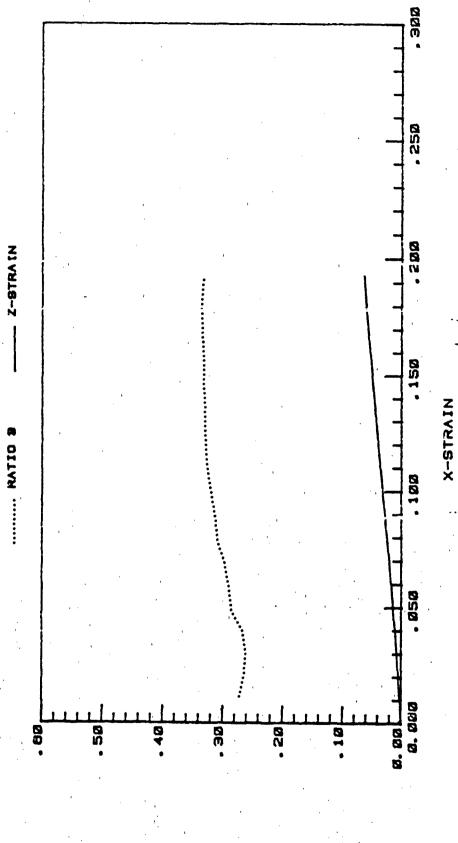


A1-27

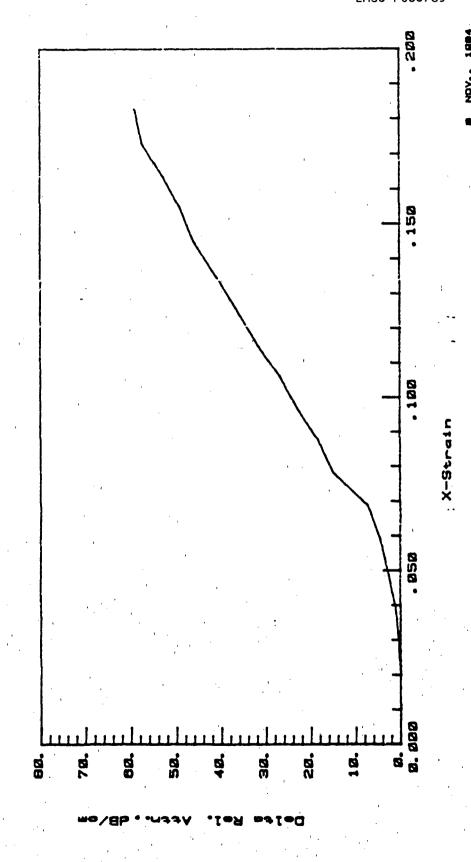
CHINA LAKE PROP. BLX-9 #0011848 1.22H X.575WX .230T 9-12-84 LOCKHEED MATERIALS ANALYSIS LABORATORY



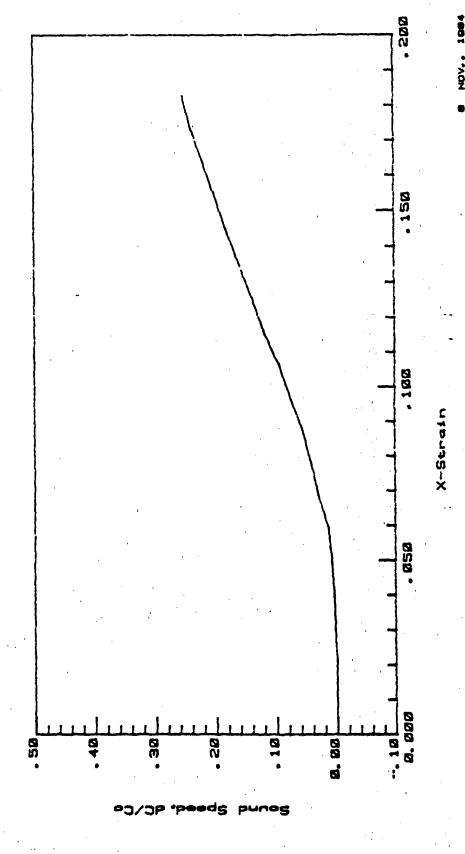
-DCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE PROP. BLX-9 #A110284 1.05H X.750 X .277T 11-02-84



CHINA LAKE PROP. BLX-0 #A110284 1.05H X.750 X .277T 11-02-64 LOCKHEED MATERIALS ANALYSIS LABORATORY

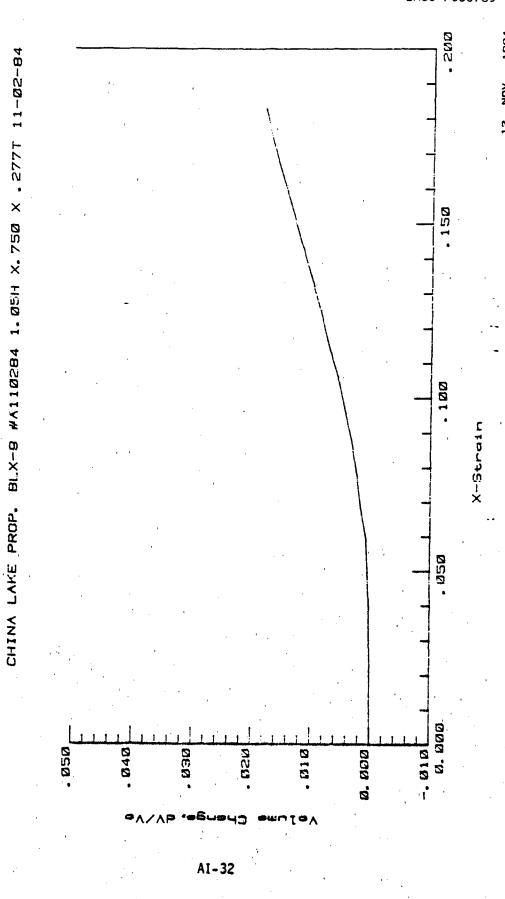


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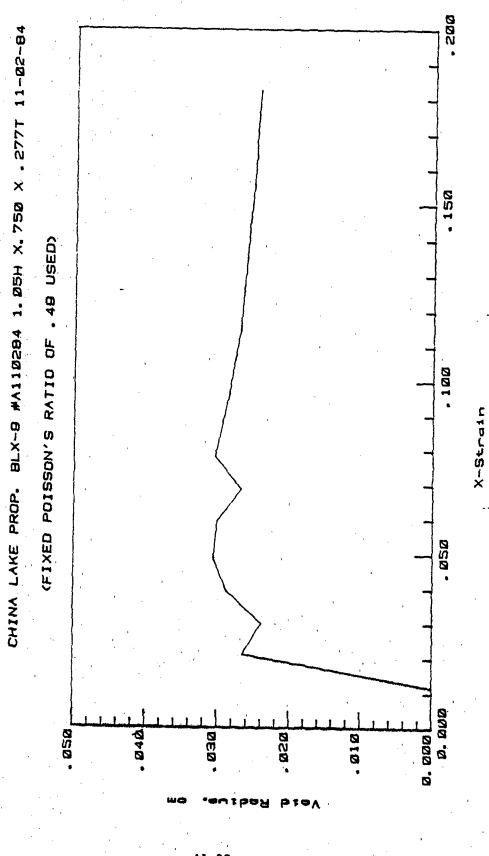


AI-31

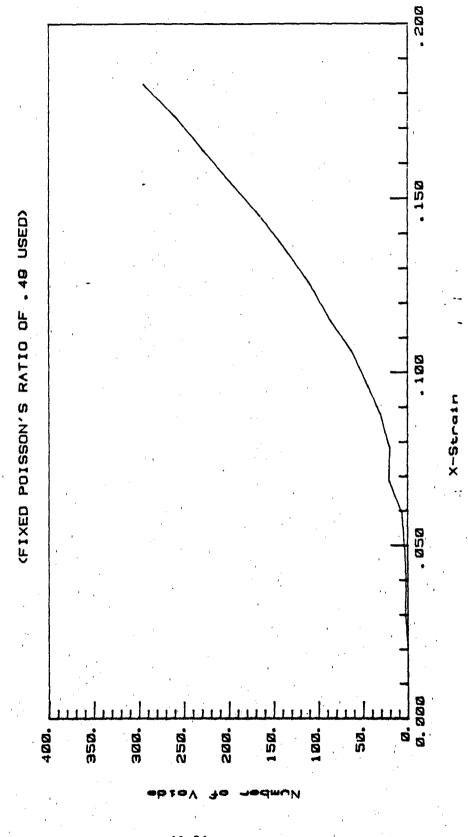
LOCKHEED MATERIALS ANALYSIS LABORATORY



COCKHEED MATERIALS ANALYSIS LABORATORY

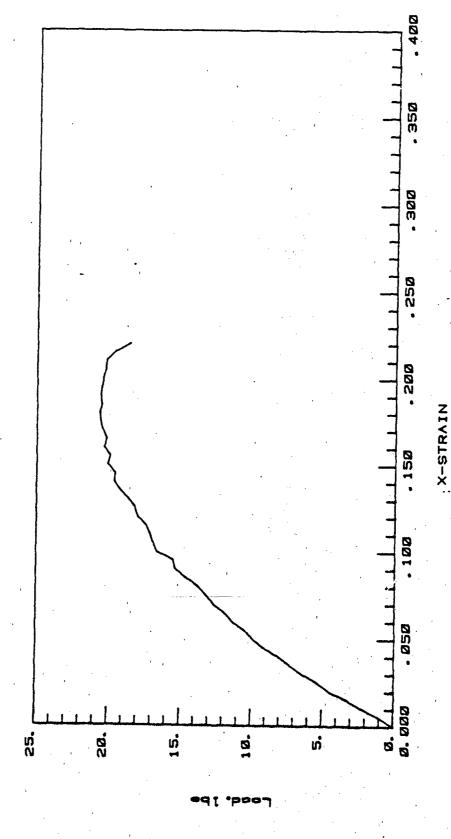


CHINA LAKE PROP. BLX-8 #A110284 1.05H X.750 X .277T 11-02-84 LOCKHEED MATERIALS ANALYSIS LABORATORY



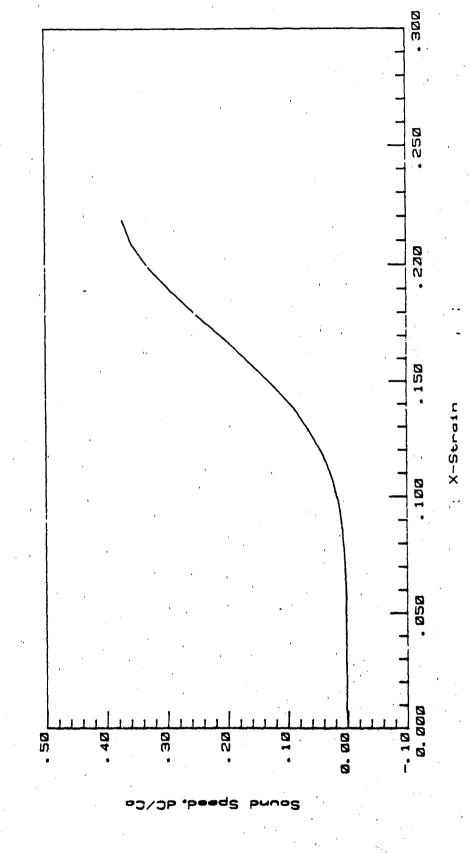
ULTRASONIC DATA FOR BLX-11

LOCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE PROPELLANT BLX-11 1H 1W . 268T A11Ø584



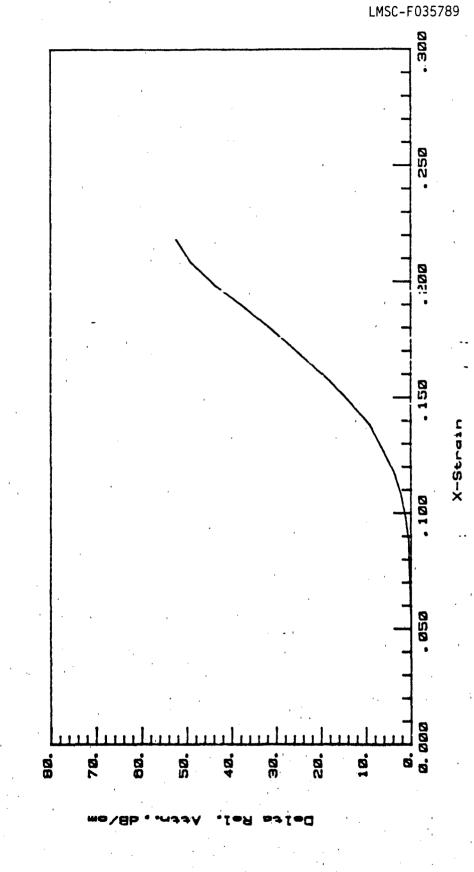
AI-36

CHINA LAKE PROP. BLX-11#A110584 1. 00514X. 1. 00X . 268T 11-05-84 LOCKHEED MATERIALS ANALYSIS LABORATORY



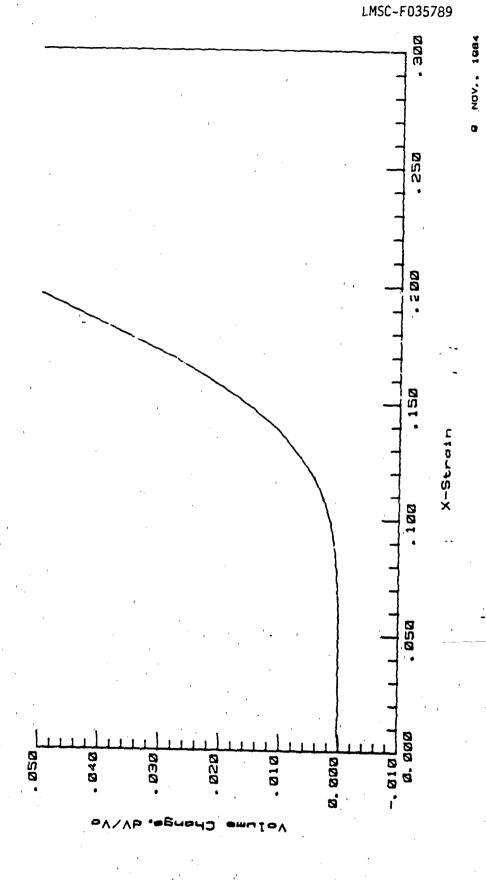
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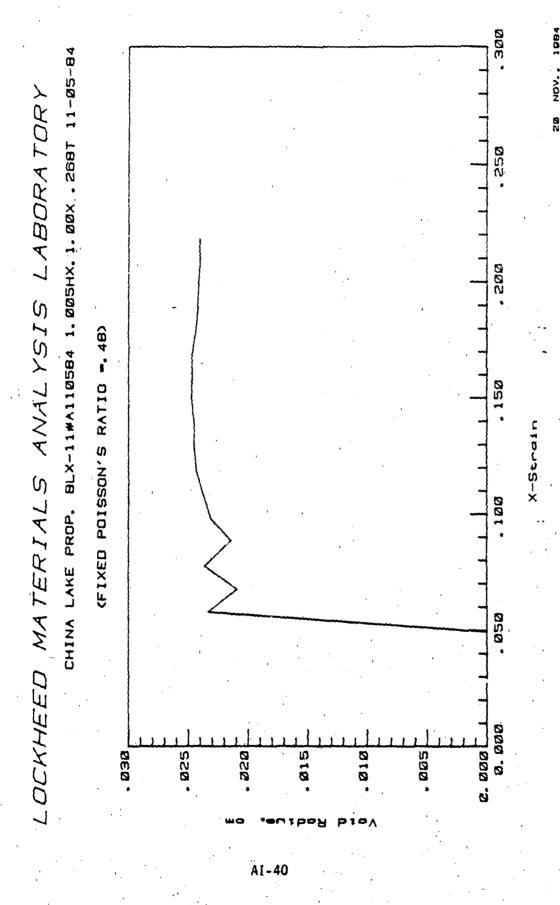
CHINA LAKE PROP. BLX-11#A110584 1.005HX. 1.00X . 268T 11-05-84



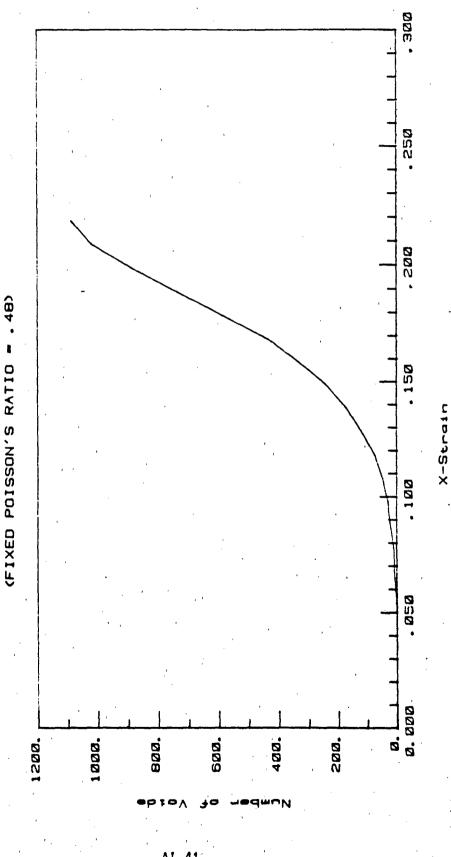
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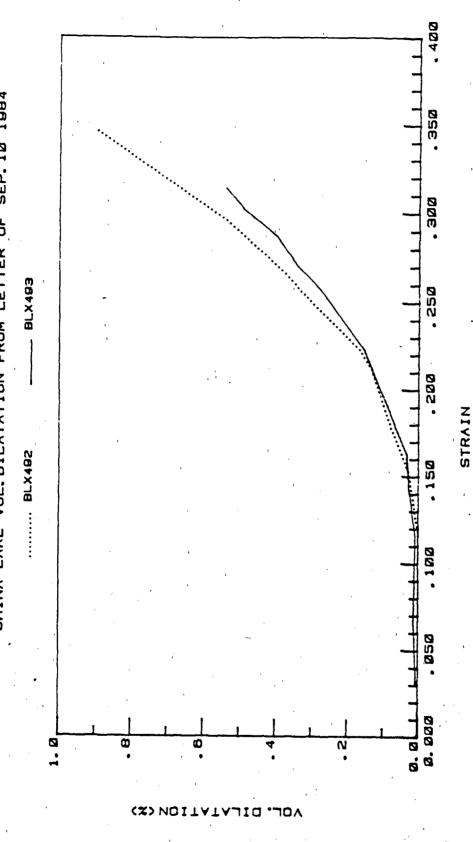
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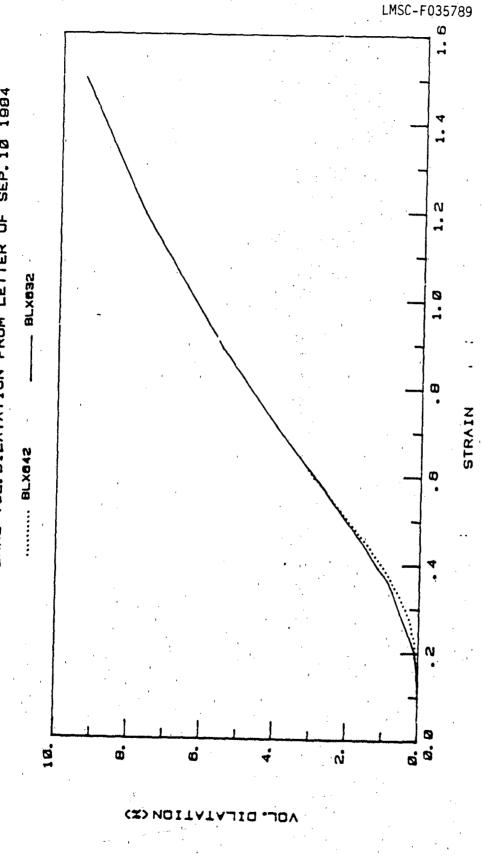
LMSC-F035789

DILATATION DATA FOR BLX-4, BLX-6, BLX-8, BLX-9, AND BLX-11(3)

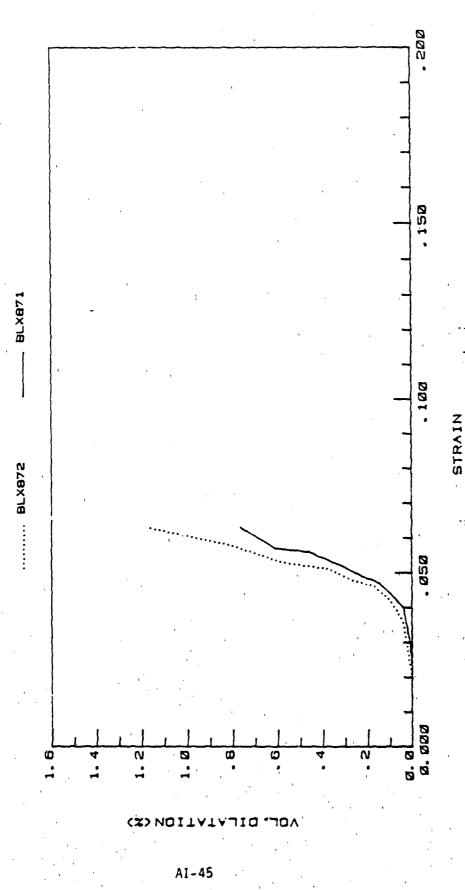
LOCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE VOL. DILATATION FROM LETTER OF SEP. 10 1984



LOCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE VOL. DILATATION FROM LETTER OF SEP. 10 1984

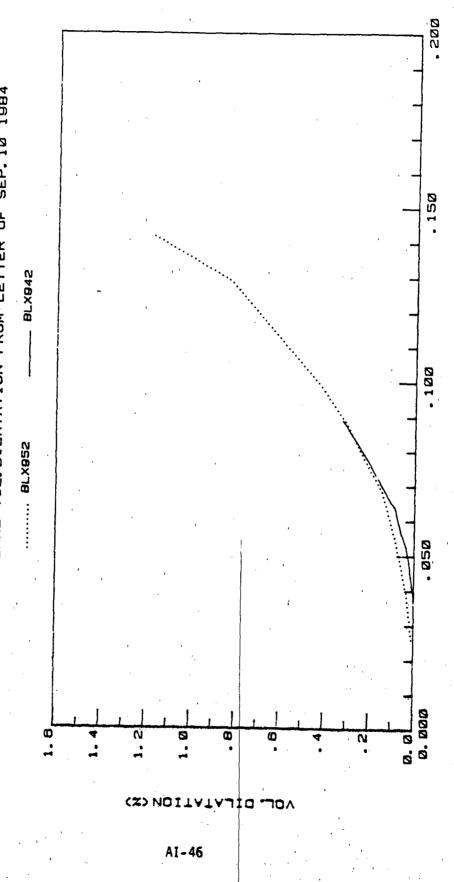


CCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE VOL. DILATATION FROM LETTER OF SEP. 10 1984

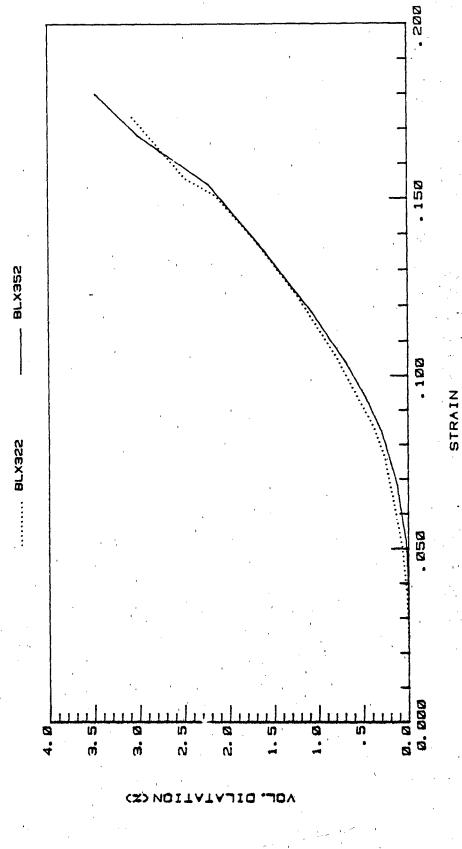




MATERIALS ANALYSIS LABORATORY CHINA LAKE VOL. DILATATION FROM LETTER OF SEP. 10 1984



LOCKHEED MATERIALS ANALYSIS LABORATORY CHINA LAKE VOL. DILATATION FROM LETTER OF SEP. 10 1984



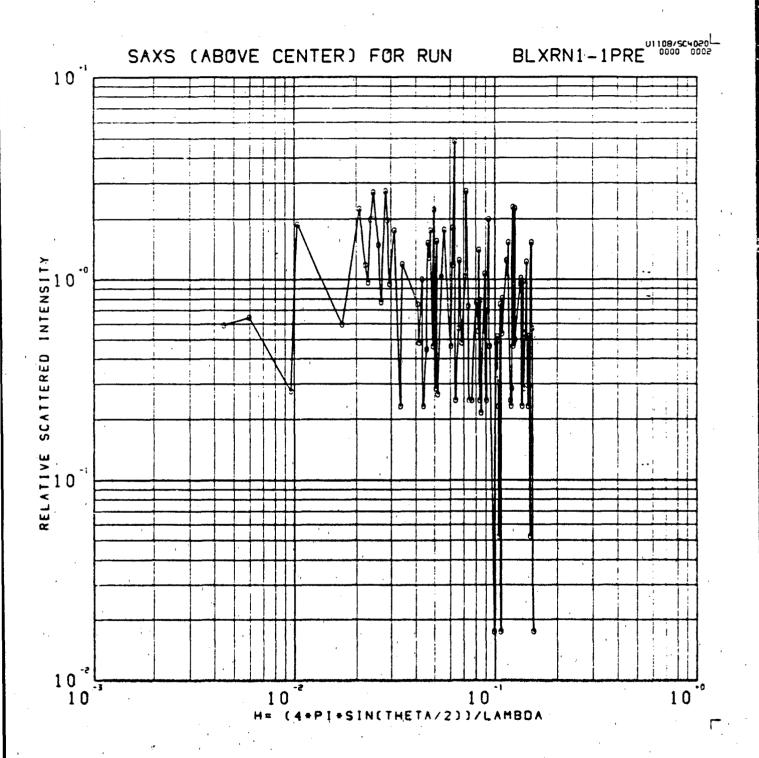
RESULTS OF ULTRASONIC MEASUREMENTS OF POISSON'S RATIO FOR 5 MATERIALS

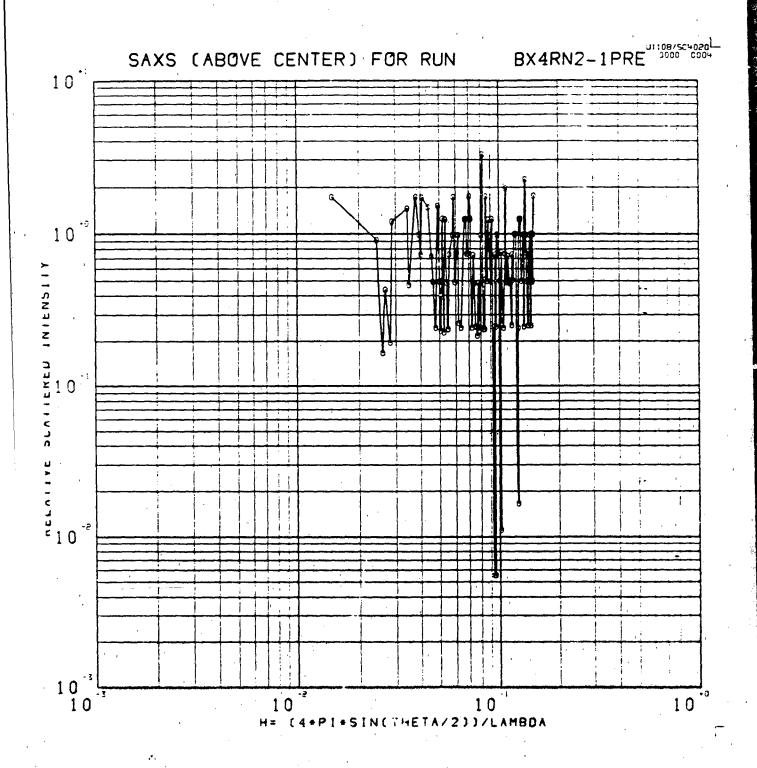
MATERIAL NUMBER	LONGITUDINAL WAVE SPEED (cm/sec) x 10 ⁻⁵	TRANSVERSE WAVE SPEED (cm/sec) x 10 ⁻⁴	POISSON'S RATIO
BLX-4	1.98	4.1	0.48
BLX-6	2.1	3.0	0.49
BLX-8	2.3	5.1	0.48
BLX-9	1.94	3.1	0.49
BLX-11	1.84	4.0	0.48

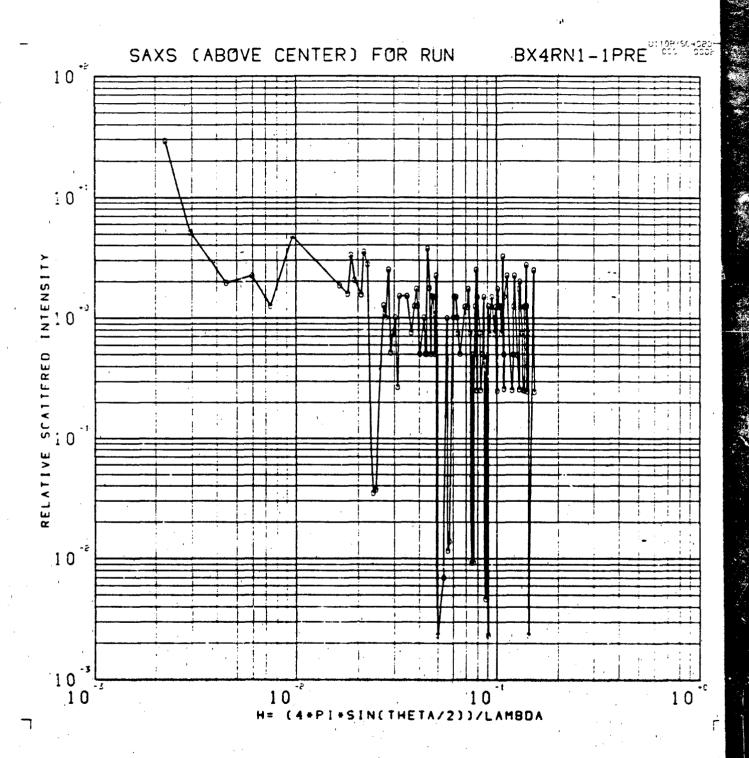
 $v_0 = \frac{1 - 0.5r^2}{1 - r^2}$

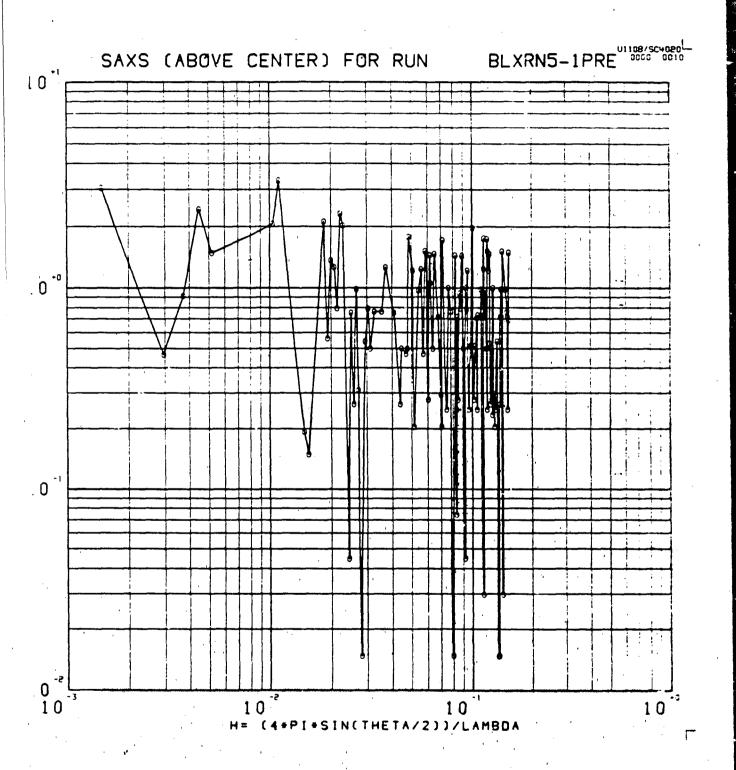
where r = IongItudinal wave speed

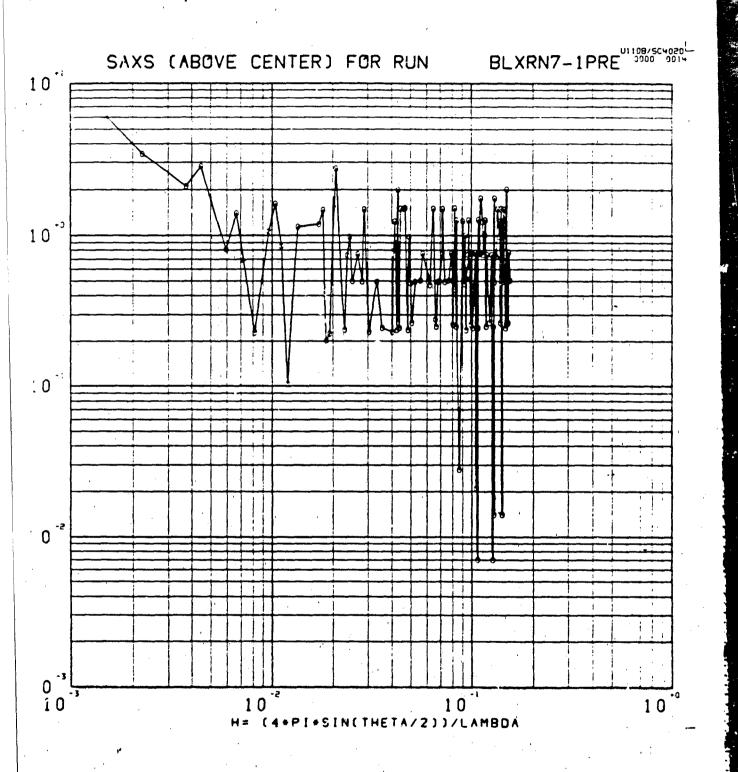
APPENDIX II
SAXS DATA FOR BLX-4

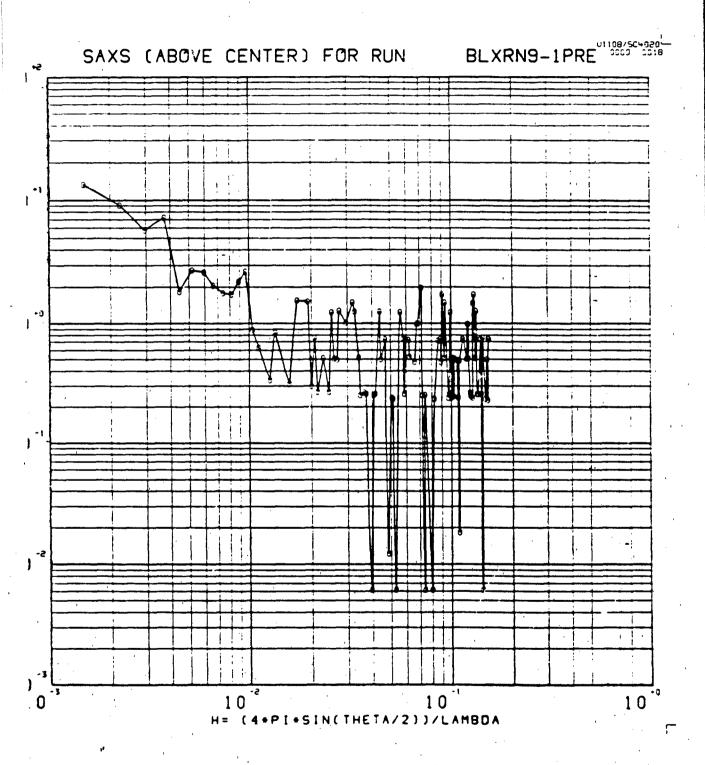


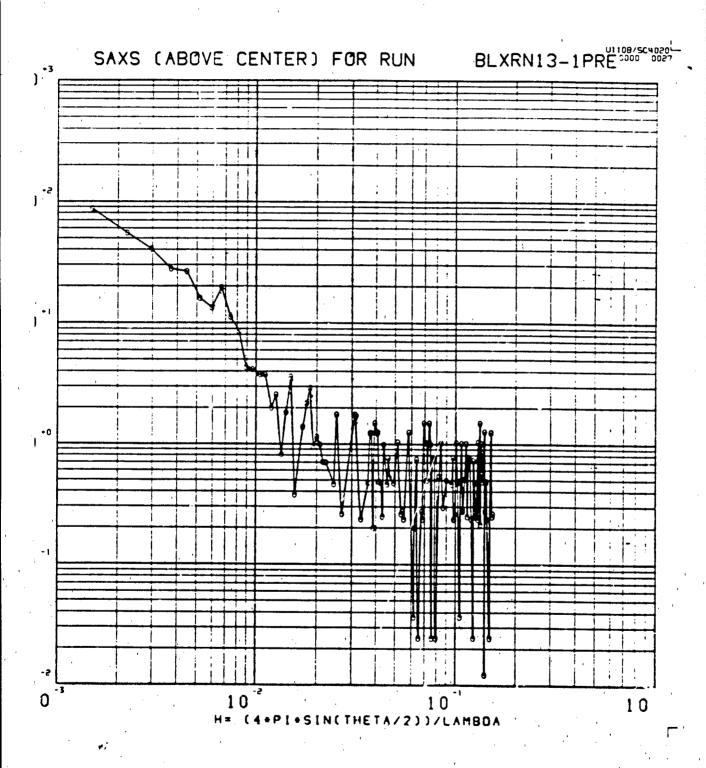


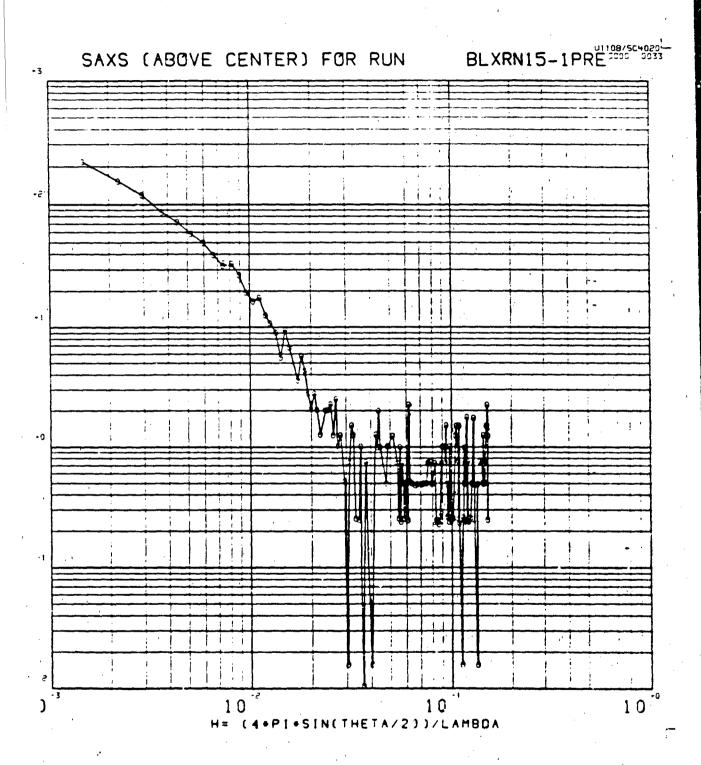


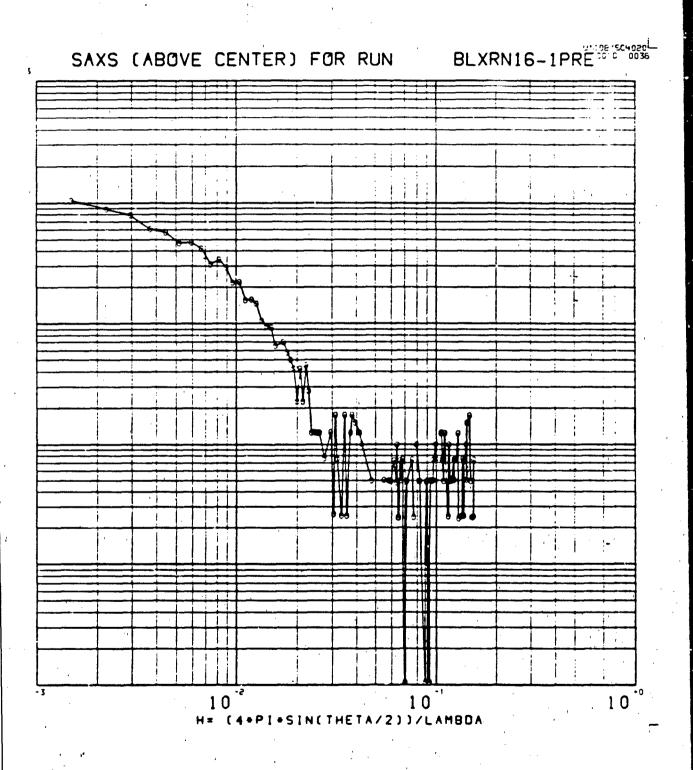


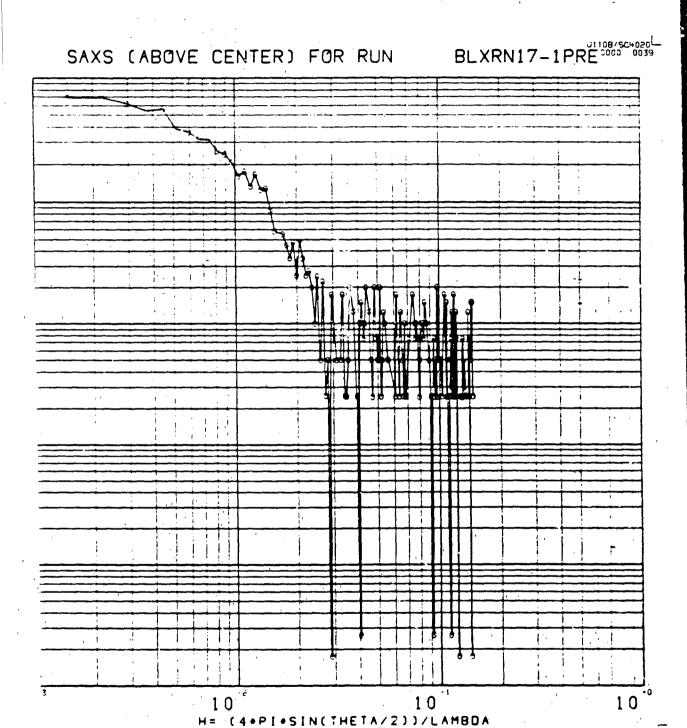




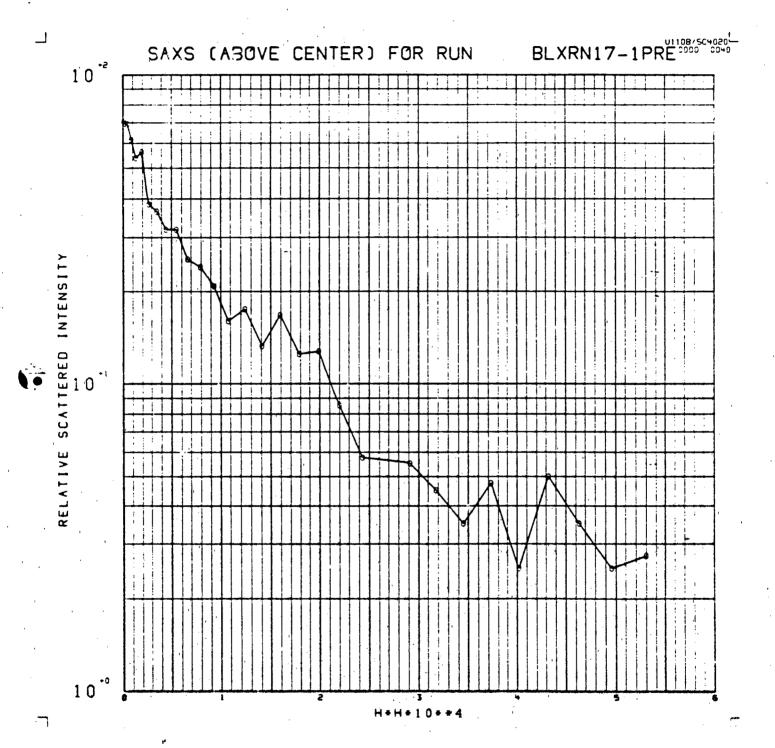


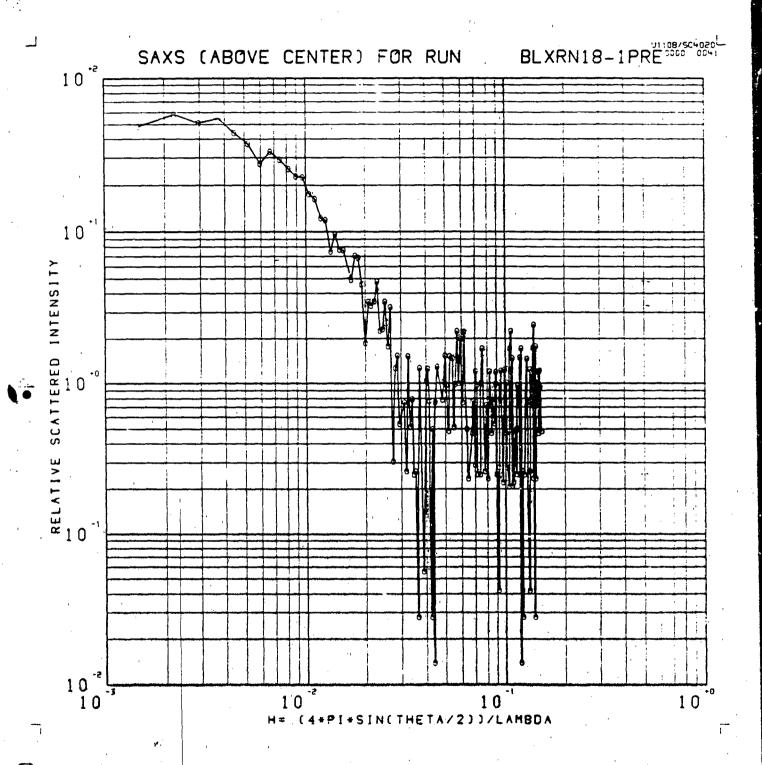




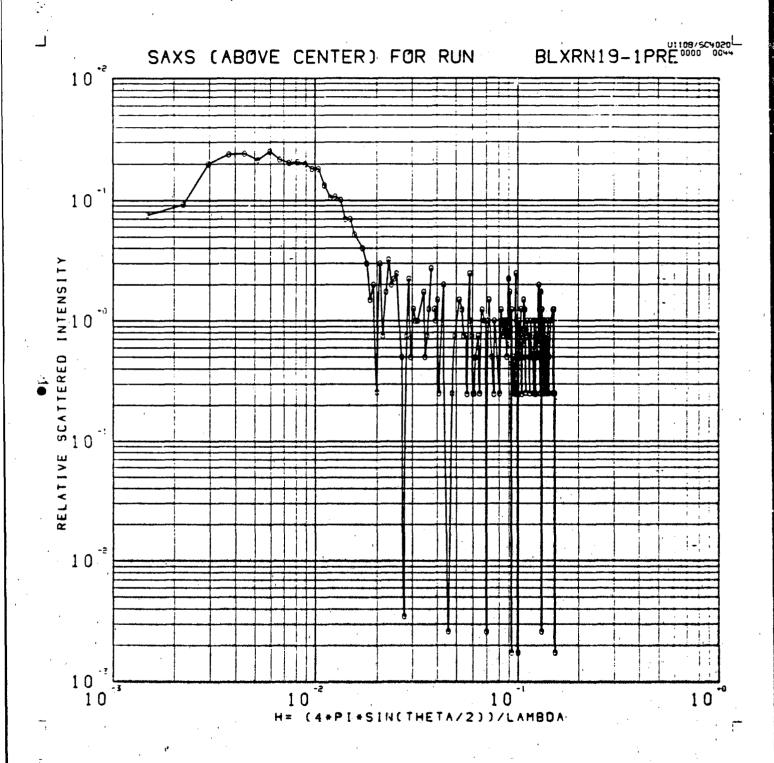


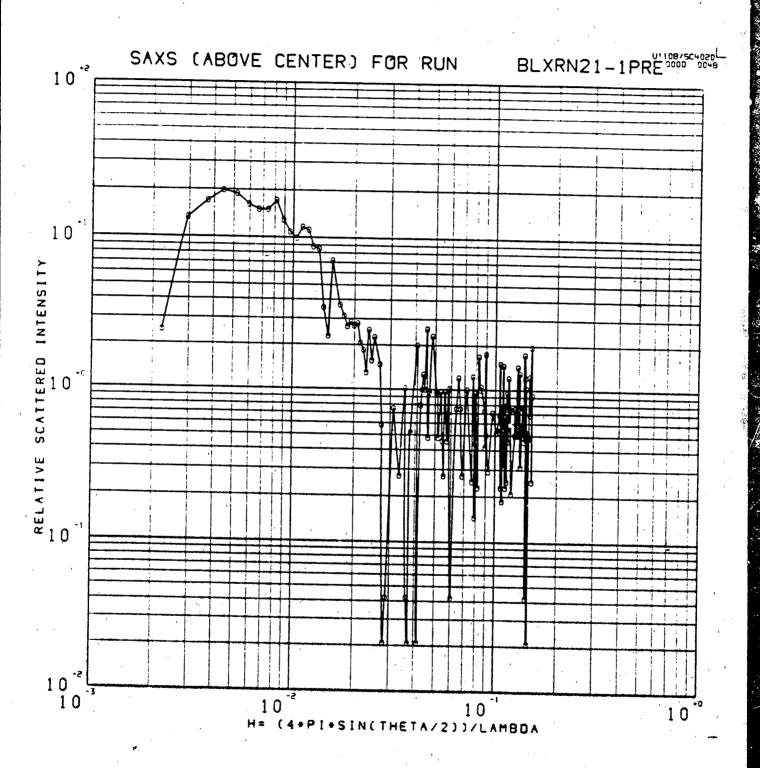
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